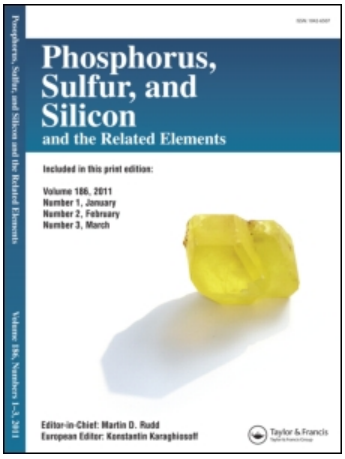

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

^{31}P and ^{13}C NMR Investigations of a Tert Butylcalix[4]Arene-Diphosphate

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To cite this Article Costisella, Burkhard and Gloede, Joerg(1996) '^{31}P and ^{13}C NMR Investigations of a Tert Butylcalix[4]Arene-Diphosphate', Phosphorus, Sulfur, and Silicon and the Related Elements, 111: 1, 19
To link to this Article: DOI: 10.1080/10426509608054648
URL: http://dx.doi.org/10.1080/10426509608054648

Phosphorus, Sulfur, and Silicon, 1996, Vol. 111, p. 19
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^{31}P AND ^{13}C NMR INVESTIGATIONS OF A TERT.BUTYLCALIX[4]ARENE-DIPHOSPHATE

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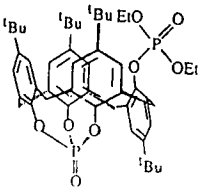
Abstract The lanthanide induced ^{31}P and ^{13}C shifts for calix[4]arenediphosphate **1** are studied. From these investigations an exact assignment of all the 48 C atoms was possible.

The aim of our investigations was to perform an exact assignment of all the 48 C atoms in the tert.butylcalix[4]arenediphosphate **1** [1]. In the ^{31}P NMR spectrum of **1** we found two signals in the typical region. It is remarkable that the shift behaviour of the cyclic and the acyclic phosphate group is very different. By addition of $Eu(FOD)_3$ the acyclic P atom shifts very strongly towards high-field but the chemical shift of the cyclic P atom is constant [2]. This behaviour is due to a steric effect of the aromatic ring with the tert.butyl substituent in the near of the cyclic P=O group. In the ^{13}C spectrum of **1** we found 34 signals, and after addition of $Eu(FOD)_3$, 36 signals. By adding the shift reagent the chemical shifts of **all** C atoms change but not the coupling constants . The shift effect is stronger in the near of the acyclic phosphorus and far from the acyclic phosphorus this effect is weaker.



partial cone form

1

An exact assignment of all the 48 C atoms can be deduced from a combination of the chemical shift values of the C atoms before and after adding $Eu(FOD)_3$, the magnitude of the coupling constants, and the peak intensities.

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